



UNIVERSITI PUTRA MALAYSIA

**FABRICATION AND CHARACTERISATION OF MONO- AND
MULTIFILAMENT Ag-SHEATHED $\text{Bi}_{1.0}\text{Pb}_{0.4}\text{Sr}_{2.0}\text{Ca}_{2.0}\text{Cu}_{3.0}\text{O}_{10}$
SUPERCONDUCTOR TAPES VIA POWDER-WIRE-IN-TUBE TECHNIQUE**

MASRIANIS BT AHMAD

FS 2005 6

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**MASTER OF SCIENCE
UNIVERSITI PUTRA MALAYSIA**

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SUPERCONDUCTOR TAPES VIA POWDER-WIRE-IN-TUBE TECHNIQUE**

By

MASRIANIS BT AHMAD

**Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia
in Fulfilment of the Requirements for the Degree of Master of Science**

December 2005



DEDICATION

*To my husband, Isa Bin Muhzan
&
my mother, Hajjah Aishah Bt Hj. Che Omar
for their love, support and understanding.*

*To my family,
for their love and concern.*

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirements for the degree of Master of Science

**FABRICATION AND CHARACTERISATION OF MONO- AND
MULTIFILAMENT Ag-SHEATHED $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$
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December 2005

Chairman: Professor Abdul Halim bin Shaari, PhD

Faculty : Science

The co-precipitation technique was used in the preparation of $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ polycrystalline ceramic superconductor powder to fabricate Ag-sheathed superconductor tapes. Powder prepared via co-precipitation method with ultra-fine grain size was used to enhance the Bi-2223 phase formation. The tapes were prepared using the powder-in-tube and powder-wire-in-tube method. The powder-wire-in-tube (PWIT) method has been developed by packing powder together with composite wires into silver tubes at the second stage of the powder-in-tube (PIT) process. Among the different routes proposed to enhance Bi-2223 phase formation, the PWIT method showed better results. The samples were prepared with different number of filaments (number of filament = 2, 4, 6, 8; PWIT and 0, 20; PIT) and different sintering times (24 hr, 48 hr and 100 hr) heated at 850 °C. Samples heated for longer time showed enhanced 2223 phase formation for pellet and monofilament tape samples from 92 % to 96 % and 84 % to 86 % respectively. The intergranular connectivity can be improved. Sintering temperature 850° enhanced the growth of the superconductor phase, which was much faster in the *ab*-plane than along the *c*-axis.

This made the platelets form well-aligned connections with each other.

in the Bi-2223 XRD peak intensity with sintering time is due to the re-arrangement of the Bi-2212 crystals, which occurs in the larger volume of liquid phase. A slight preferred orientation of the grains in the *c*-axis direction when the samples were rolled into tapes was also observed. By increasing the sintering time the average grain size is increase from 4 μm to 14 μm and by increasing the number of filaments from 2 to 8 filaments inside the tube and core area that developed the non-superconducting phase and the secondary phase were minimized. Small filaments in multifilament tapes are more homogenous than monofilament tapes owing to the better grain orientation along the silver sheath. At 77 K and zero field, the highest transport critical current density (J_C) $11500 \pm 300 \text{ A/cm}^2$ was achieved in the tape prepared via PWIT (number of filament = 8) sintered for 100 hours compared to PIT method with transport critical current density (J_C) $7800 \pm 300 \text{ A/cm}^2$ at the same sintering duration. Therefore, critical current density increases with the sintering duration and number of filaments.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

**FABRIKASI DAN PENCIRIAN MONO DAN MULTIFILAMEN PITA
SUPERKONDUKTOR $\text{Ag-Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ SARUNGAN-Ag MELALUI
TEKNIK SERBUK-DAWAI-DALAM-TIUB**

Oleh

MASRIANIS BT AHMAD

December 2005

Pengerusi : Profesor Abdul Halim bin Shaari, PhD

Faculti : Sains

Teknik pemendakan bersama telah digunakan bagi menyediakan superkonduktor seramik polihablur $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ untuk fabrikasi pita superkonduktor sarungan Ag. Serbuk disediakan dengan kaedah pemendakan bersama untuk menghasilkan serbuk bersaiz ultra-halus bagi meningkatkan pembentukan fasa Bi-2223. Pita telah disediakan menggunakan teknik serbuk-dalam-tiub dan serbuk-dawai-dalam-tiub. Teknik serbuk-dawai-dalam-tiub (PWIT) dibangunkan dengan memasukkan serbuk bersama-sama dawai ke dalam tiub Ag pada peringkat yang kedua teknik serbuk-dalam-tiub (PIT). Di antara pelbagai kaedah dalam meningkatkan pembentukan fasa Bi-2223, teknik serbuk-dawai-dalam-tiub turut memberikan keputusan yang baik. Sampel disediakan dengan bilangan filamen yang berbeza (bilangan filamen = 2, 4, 6, 8; PWIT dan 0, 20; PIT) dan masa pembakaran yang berlainan (24 jam, 48 jam dan 100 jam) pada suhu 850 °C. Sampel yang dibakar lama menunjukkan pembentukan fasa 2223 meningkat bagi sampel pelet dan pita masing-masing dari 92 % ke 96 % dan 84 % ke 86 %. Keadaan struktur butiran antara butiran diperbaiki. Pembakaran melebihi 850° meningkatkan pertumbuhan

fasa superkonduktor yang mana ia lebih cepat dalam arah ab berbanding arah c . Ini menunjukkan lapisan-lapisan disusun mudah di antara satu sama lain. Peningkatan puncak Bi-2223 dengan masa pembakaran ialah kerana hablur Bi-2212 menyusun semula menyebabkan fasa cecair bertambah. Didapati bahawa susunan butiran adalah dalam arah c bagi sampel yang telah digolek. Dengan meningkat masa pembakaran, purata saiz butiran meningkat dari $4\text{ }\mu\text{m}$ ke $14\text{ }\mu\text{m}$ dan dengan menambah bilangan filamen dari 2 ke 8 filamen ke dalam tiub Ag menyebabkan kawasan tengah yang di pelopori oleh fasa bukan superkonduktor dan fasa sekunder dapat di kurangkan. Filamen halus dalam pita multifilamen adalah lebih homogen berbanding monofilamen disamping menghasilkan susunan butiran yang lebih baik di sepanjang permukaan perak. Pada suhu cecair nitrogen, 77 K dan tanpa medan, ketumpatan angkutan arus genting (J_C) ialah $11500 \pm 300\text{ A/cm}^2$ dicapai bagi pita yang disediakan menggunakan teknik serbuk-dawai-dalam-tiub (Mu08100) berbanding sampel pit100 yang menggunakan teknik serbuk-dalam-tiub dengan ketumpatan angkutan arus genting (J_C) ialah $7800 \pm 300\text{ A/cm}^2$. Oleh itu, ketumpatan angkutan arus genting (J_C) meningkat dengan tempoh masa pembakaran dan bilangan filamen.

ACKNOWLEDGEMENTS

I am extremely grateful to my supervisor, Professor Dr. Abdul Halim Shaari chairman of supervisory for most of all, believing in me and for his invaluable advice, patience, guidance, ideas, criticism, encouragement and continuous discussion. My deepest gratitude goes to my co-supervisors, Professor Dr. Roslan Abdul Shukor, Universiti Kebangsaan Malaysia and Associate Professor Dr. Mansor Hashim, Universiti Putra Malaysia, members of the supervisory committee for the positive assistance, comments, suggestions and wise guidance throughout the research work.

I am very grateful for the financial assistance provided through the Intensified Research Program in Priority Area (IRPA) and PASCA. My special thanks go to Dr. Imad Hamadneh, Mr Syed Yusainee Syed Yahya and Mr Lau Kok Tee for their guidance, suggestion and support.

The assistance provided by the Department of Physics UPM for the use of their X-ray diffraction facility, Institute of Bioscience UPM for the use of their SEM facility; to Associate Prof. Dr. Fauziah Othman, Mr. Ho, Miss Azilah, Mrs. Faridah and all members of Electron Microscopy Unit, thanks a lot for your kind assistance and the librarians of UPM and UKM, is gratefully acknowledgment.

I am extremely grateful to my lab mates, Dr. Lim Kean Pah, Dr. Abdullah Chik, Dr. Kabashi, Huda Abdullah, Lee Oon Jew, Mustafa Awang Kechik, Priscilla Ibai, Mohd Faisal Mohd Aris and Walter Charles Primus. Thanks a lot for their tremendous



assistance and support throughout this study. To my friends who never fail to encourage me at UKM until the end; Puan Hajjah Rokiah Mohd Yasin, Puan Norazilah, Miss Yip, Mr. Lee Chee Hong, Mr David and Mr Lee Teik Huye, my special thanks go to all of you. I am very thankful to Mr Razak Harun, Mr. Razi, and other technical staff in the Physics Department for their technical favours.

To my late father, my mother, brothers; Mr Udanis Ahmad and his wife, Mr Adam Ahmad, Mr Ustazanis Ahmad and Mr Al-Qurannis and my sister, Mrs Surianis Bt Ahmad, their love and support keep me going, Last but not least, to my husband, Isa Bin Muhzan, thank you for your love, continuous support, encouragement and understanding.


I certify that an Examination Committee has met on 23rd December 2005 to conduct the final examination of Masrianis Bt Ahmad on her Master of Science thesis entitled “Fabrication And Characterisation of Mono- And Multifilament Ag-Sheathed $\text{Bi}_{1.6}\text{Pb}_{0.4}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_{10}$ Superconductor Tapes via Powder-Wire-In-Tube Technique” in accordance with Universiti Pertanian Malaysia (Higher Degree) Act 1980 and Universiti Pertanian Malaysia (Higher Degree) Regulations 1981. The Committee recommends that the candidate be awarded the relevant degree. Members of the Examination Committee are as follows:

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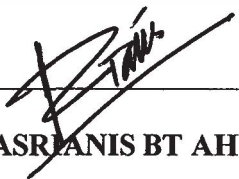
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Date: **13 APR 2006**

DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.



MASRIANIS BT AHMAD
Date: 20/3/2006

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ABBREVIATIONS AND KEY WORDS

BCS theory	Bardeen, Cooper and Schrieffer theory
LBCO	La-Ba-Cu-O system
YBCO	Y-Ba-Cu-O system
Y123	Family member in $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$.
Bi-2201	Family member in $\text{Bi}_2\text{Sr}_2\text{Ca}_n\text{Cu}_{n+1}\text{O}_{6+2n}$, $n=0$
Bi-2212	Family member in $\text{Bi}_2\text{Sr}_2\text{Ca}_n\text{Cu}_{n+1}\text{O}_{6+2n}$, $n=1$
Bi-2223	Family member in $\text{Bi}_2\text{Sr}_2\text{Ca}_n\text{Cu}_{n+1}\text{O}_{6+2n}$, $n=2$
TBCCO	Tl-Ba-Ca-Cu-O system
Tl-2223	Family member in $\text{Tl}_2\text{Ba}_2\text{Ca}_n\text{Cu}_{n+1}\text{O}_{6+2n}$, $n=2$
HBCCO	Hg-Ba-Ca-Cu-O system
TGA	Thermo Gravimetric Analysis
DTA	Differential Thermal Analysis
Calcination	Heating process where the solid state reaction occur
Sintering	Heating process yielding for more compacting of the sample grains and improve its properties
PIT	Powder-in-tube
PWIT	Powder-wire-in-tube
B , H	Magnetic Field
B_{\parallel}	Magnetic field parallel to tape face
B_{\perp}	Magnetic field perpendicular to tape face
B_c	Critical magnetic field
B_{c1}	Lower Critical Field
B_{c2}	Upper Critical Field
T	Temperature
T_c	Critical Temperature
$T_{c\text{-onset}}$	Critical Temperature onset
$T_{c\text{-zero}}$	Critical Temperature zero
I	Current
E-I	Electric Field - Current
A	Surface area
V	Voltage